

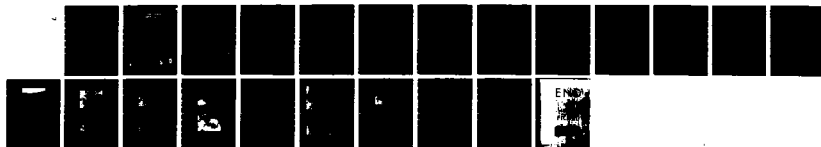
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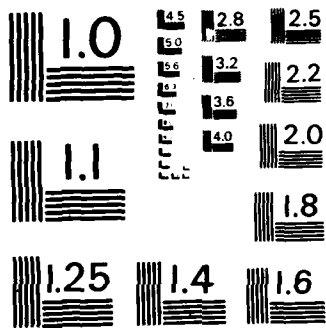
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# APPLICATION OF RAPIDLY SOLIDIFIED ALLOYS

A. R. Cox  
United Technologies Corporation  
Pratt & Whitney Aircraft Group  
Box 2691, West Palm Beach, Florida 33402

December 1978  
Quarterly Report for Period 1 August 1978 Through 1  
November 1978

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Prepared for  
Air Force Materials Laboratories  
Wright-Patterson AFB, Ohio 45433

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)  This program is being conducted for the purpose of applying the principle of rapid solidification to aluminum and iron alloy powders and subsequent development of stronger alloy compositions for fan blade application (Al alloys) and higher speed bearing material (Fe alloys). Centrifugal atomization and forced convective cooling are being used to produce the fast cooled powder. During this report period, adaptation of the RSR process to aluminum and iron systems was continued. Both Al and Fe alloys were produced and the Al alloys were consolidated by direct extrusion. Hardness and mechanical testing were begun for initial evaluation of aluminum alloys.		

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## SECTION I

### INTRODUCTION

Rapid solidification of metal alloys has shown that distinct and dramatic changes in microstructure and crystal form can be attained beyond those possible using any conventional method of solidification. These results are recognized by experts throughout the field of metallurgy as a means to achieve major improvements in metal strength, environmental compatibility, electrical properties, etc. Through the use of fast cooling, the following appears to be eminently possible: (1) stronger and more corrosion resistant steels because of improved homogeneity and (2) a new breed of aluminum, copper, and nickel alloys because of improved secondary phase dispersion.

An Advanced Research Project Agency (ARPA) sponsored program with the Pratt & Whitney Aircraft Group, Government Products Division (P&WA/Florida), has shown that by using the P&WA RSR process and equipment, it is possible to achieve rapid solidification in spherical powder under conditions which depict steady-state operations commensurate with production rates in excess of 1400 lb/hr. Further, this program has demonstrated that concurrent high product quality can be achieved and the resulting powder metal is in a form which can be readily handled and processed into useful shapes for subsequent application. No other method known to achieve similar rates of solidification can lay claim to these combined achievements.

The program has gone even further since it has demonstrated that controlled, rapid solidification can lead to a microcrystalline form, a condition which could possibly point the way to alloy homogeneity never before considered possible. It has also shown that a central rotary source can be used for liquid metal atomization into powder particles of sizes commensurate with average particle cooling rates of  $10^4 - 10^6$  °K/sec.

This program is a modification to the ARPA sponsored work which is directed toward superalloy development. Its purpose is to expand the scope of work in the field of rapid solidification from the exclusive study of superalloys to a study of aluminum (Al) and iron (Fe) base alloys. The specific objectives of this added effort are the development of an improved aluminum alloy suitable for V/STOL-A fan blades and an improved iron alloy suitable for rolling element bearings for advanced aircraft powerplants.

The program is a 36-month effort which begins with adaptation of the rapid solidification rate process to Al and Fe alloy systems and terminates with a payoff analysis of new materials as adapted to V/STOL-A and F100 advanced engine derivative requirements. This is the second technical report and covers the fourth through the sixth months of the program. It deals with adaptation of RSR processing to Al and Fe systems and the subsequent evaluation of these alloys.

## SECTION II

### MATERIAL SELECTION

Several alloy selections were added to the first Al and Fe matrices, as shown in Table 1. The emphasis was placed on Al alloys, concentrating on the higher Zn contents, 8.4 to 9.8% and covering the full range of Co concentrations 0.8 to 3.2%.

TABLE 1. FIRST Al AND Fe MATRIX

Al Matrix*					
Cobalt					
Zn	0.8	1.6	2.4	3.2	
5.6	X	X		X	
7.0	X				
8.4	X	X		X	
9.8	X		X		
Fe Matrix*					
Molybdenum					
Cr	C	2	4	6	8
4	0.8		X (M-50)		
9	0.8				
	9.5				
14	0.8				
	1.1		X (EX-00007)	X	
19	0.8				
	1.25	X			X

1% V held constant

\*Amounts are in wt %

Co additions form an intermetallic compound  $\text{Co}_3\text{Al}$ , which is insoluble up to the solidus temperature of the alloy. If present in a fine dispersion the  $\text{Co}_3\text{Al}$  is expected to control grain size and provide dispersion strengthening. High Zn contents should provide additional second phase material for precipitation hardening.

The addition to the steel matrix was M-50 steel. This will provide a comparison of the effects of rapid solidification to conventionally processed material.

Fe and Al experimental alloy samples were produced in the form of buttons weighing 2 oz (60 gm) for Fe and 1 oz (30 gm) for Al. A computer program was developed which makes random alloy selections, when the input data are desired alloying elements and their range of compositions. Thirty experimental Fe alloys were melted by a tungsten arc melting device and treated by electron beam surface irradiation to produce macroregions depicting rapid solidification. Various compositions are shown in Table 2. After difficulty in obtaining proper alloying, a method was devised to make experimental Al alloys with desired compositions by induction melting in small ceramic crucibles. Iron alloys were produced in the arc melting device and no problems were encountered in alloying.

During the next quarter the first Al matrix will be repeated; however, the powder will be handled inertly rather than in air as was the case previously.



TABLE 2. COMPOSITION OF EXPERIMENTAL IRON ALLOYS

<i>Alloy No.</i>	<i>CR</i>	<i>MO</i>	<i>V</i>	<i>Mn</i>	<i>Ni</i>	<i>C</i>	<i>Fe</i>
1	16.262	1.3045	2.2628	0.99374	1.2795	1.3172	76.581
2	16.282	6.1318	2.1869	1.1752	1.188	1.3332	71.703
3	13.851	3.9705	1.8493	1.1467	1.0661	1.0685	77.048
4	10.055	6.1623	0.72107	0.47221	0.93889	0.82412	80.826
5	15.354	6.1268	2.2371	0.7288	0.92113	1.4247	73.207
6	10.624	6.8809	1.4754	0.98778	0.71623	0.77248	78.543
7	14.846	5.5949	0.68559	0.70239	1.3789	1.0426	75.75
8	11.019	2.6297	2.1411	0.5336	1.1982	1.2464	81.232
9	17.321	0.31295	0.7745	0.5433	1.0239	1.1169	78.908
10	15.479	7.0256	1.4145	1.1583	0.44825	0.83952	73.637
11	14.751	4.98	2.1762	0.33929	0.37178	0.82849	76.553
12	17.85	7.386	1.6684	0.36571	0.62287	0.7828	71.324
13	12.584	6.513	2.1626	1.1352	0.86522	1.3285	75.412
14	15.928	2.467	0.73184	0.48411	0.41893	1.0239	78.946
15	17.674	7.3168	0.59652	0.67471	1.2361	0.84584	71.656
16	12.737	0.51408	0.53022	0.86849	0.71243	0.95239	83.685
17	10.656	1.3654	2.1569	0.88963	0.68281	0.98525	83.264
18	11.739	6.6256	2.3339	1.3537	0.43752	0.76429	76.746
19	13.752	0.097094	1.5491	0.28522	0.31381	1.1199	82.883
20	12.991	4.4284	2.367	0.86825	0.55627	0.86252	77.926
21	13.016	1.1215	1.449	0.77303	0.306	0.87249	82.462
22	17.234	1.0864	1.5247	0.37111	0.36896	1.3236	78.111
23	14.021	0.73855	0.9322	0.90031	1.0294	1.0865	81.292
24	12.919	4.5009	0.95091	1.2614	0.41511	1.0514	78.901
25	13.979	7.2712	2.1363	0.22644	0.78698	0.82102	74.779
26	17.172	2.1241	1.2243	1.2092	1.0151	0.88972	76.365
27	17.297	4.2095	0.90901	1.1961	0.82059	1.1718	74.396
28	17.698	1.685	0.90469	1.3583	1.1022	1.1873	76.065
29	13.578	4.1678	1.1898	0.85955	0.63201	1.0615	78.511
30	14.619	3.5277	2.1878	0.45148	0.79586	1.1749	77.244

### SECTION III

#### CONVERSION AND CONSOLIDATION

Twelve powder runs were attempted during the second quarter to gain more experience in operation of the AGT 500,000 and to provide additional material for evaluation. Nozzle diameter, cup radius and pour temperature were varied to assess their impact on powder yield. Some difficulties were encountered and are under investigation to assure the best possible yield of -140 mesh (105 micron) powder. Tables 3 and 4 give material compositions, pertinent information, and comments on the twelve conversions.

Eleven XSR Al conversions were attempted with seven being successful. The yields in each case were in excess of 50%.

Four XSR runs did not pour due to metal solidifying in the nozzle. This problem was caused by a lack of superheat which allowed segregation of a Co-Al phase within our vacuum melt master heats. Pour temperatures used in the AGT 500,000 were not sufficient to completely melt the Co-Al phase which settled to the bottom of the tundish. As a result, the first material poured was a liquid solid slush which solidified upon reaching the nozzle area. This situation was corrected by modifying vacuum melt procedures to prevent segregation and apply additional superheat during XSR conversions.

The first conversion attempted in a redesigned high-temperature furnace was XSR-46. This furnace incorporates induction melting with a tantalum resistance heater surrounding the nozzle to provide a separate nozzle temperature control thereby preventing solidification due to low nozzle temperature.

Although XSR-46 was successful, the yield was low, 26.3%, due to a malfunctioning helium quench valve which occurred during the first half of the conversion. This situation was corrected and the expectation is that yields from steel runs will be comparable to Al runs.

Twelve Al extrusions were completed at AFML, which covered seven compositions and three extrusion temperatures, 700°F (371°C), 750°F (399°C) and 800°F (427°C). The reduction ratio was 16:1 through a 60 deg included angle steel die. Final diameter was a nominal 0.75 in. (1.9 cm). Tables 5 and 6 give particulars on each extrusion.

Three alloys XSR 30-1, 33, and 36 were selected from the Al extrusions for further reduction by swaging. A reduction ratio of approximately 3.5:1 was used to give a nominal diameter of 0.40 in. (1 cm). Swaging took place at 750°F (399°C). Seven passes were necessary with reheating between each to achieve final diameter.

Table 7 lists the reduction schedule for XSR 30-1, which is typical of the other two alloys swaged.

TABLE 3. POWDER RUNS ATTEMPTED DURING SECOND QUARTER

XSR Run No.	Alloy	Nozzle Diameter in. (cm)	Cup Speed, rpm	Cup Radius in. (cm)	Nozzle Temperature, °F (°C)	Pour Temperature, °F (°C)	Alloy Melting Point, °F (°C)	Yield, <sup>a</sup> %	Comments
XSR-34	VM 616	0.100 (0.254)	24K	3.125 (7.938)	1250 (677)	1500 (816)	1160 (627)	0	Solidified in Nozzle
XSR-35	VM 615	0.100 (0.254)	24K	3.125 (7.938)	1240 (671)	1500 (816)	1160 (627)	71.0	
XSR-36	VM 617	0.100 (0.254)	24K	3.125 (7.938)	1250 (677)	1500 (816)	1170 (632)	54.0	
XSR-37	VM 617	0.100 (0.254)	24K	3.125 (7.938)	1210 (654)	1460 (793)	1160 (627)	63.5	
XSR-38	VM 616	0.100 (0.254)	24K	3.125 (7.938)	1240 (671)	1500 (816)	1160 (627)	0	Solidified in Nozzle
XSR-39	VM 622	0.100 (0.254)	24K	3.125 (7.938)	1250 (677)	1500 (816)	1150/1160 (621/627)	0	Solidified in Nozzle
XSR-40	VM 622	0.100 (0.254)	24K	3.125 (7.938)	1300 (704)	1600 (871)	1160 (627)	66.5	
XSR-41	VM 622	0.100 (0.254)	24K	5.250 (13.335)	1290 (699)	1600 (871)	1160 (627)	0	Solidified in Nozzle
XSR-43*	VM 630	0.125 (0.318)	24K	3.600 (9.144)	1480 (804)	1760 (960)	1160 (627)	50.9	
XSR-44	VM 630	0.125 (0.318)	24K	3.600 (9.144)	1300 (704)	1500 (816)	1160 (627)	70.8	
XSR-45	VM 631	0.125 (0.318)	24K	3.600 (9.144)	1320 (716)	1500 (816)	1160 (627)	60.3	
XSR-46	VM 649	0.125 (0.318)	24K	3.600 (9.144)	2450 (1343)	2700 (1482)	2430 (1332)	26.3	No Helium Quench 1st Half of Run

<sup>a</sup>Percent Yield = weight of - 140 Mesh/wt charged.

\*XSR-43 and all subsequent runs have inert handling of powder.

TABLE 4. COMPOSITION OF ALLOYS CONVERTED TO POWDER\*

<i>Powder Run No.</i>	<i>VM No.</i>	<i>Zn</i>	<i>Co</i>	<i>Al</i>	<i>Mg</i>	<i>Cu</i>	<i>Cr</i>	<i>Mo</i>	<i>C</i>	<i>V</i>	<i>Fe</i>
XSR-34	616	8.4	0.8	Bal	2.5	1.0					
XSR-35	615	7.0	3.2	Bal	2.5	1.0					
XSR-36	617	8.4	1.6	Bal	2.5	1.0					
XSR-37	617	8.4	1.6	Bal	2.5	1.0					
XSR-38	616	8.4	0.8	Bal	2.5	1.0					
XSR-39	622	8.4	3.2	Bal	2.5	1.0					
XSR-40	622	8.4	3.2	Bal	2.5	1.0					
XSR-41	622	8.4	3.2	Bal	2.5	1.0					
XSR-43	630	9.8	0.8	Bal	2.5	1.0					
XSR-44	630	9.8	0.8	Bal	2.5	1.0					
XSR-45	631	9.8	2.4	Bal	2.5	1.0					
XSR-46	649	—	—	—	—	—	4.0	4.0	0.8	1.0	Bal

\*wt %

TABLE 5. ALUMINUM EXTRUSION PARAMETERS

<i>Billet ID No.</i>	<i>Alloy</i>	<i>Temperature °F (°C)</i>	<i>Tonage*</i>
XSR-19	VM 578	700 (371)	270
XSR-20	VM 578	750 (399)	215
XSR-23	VM 581	750 (399)	240
XSR-25	VM 587	750 (399)	240
XSR-27	VM 583	750 (399)	235
XSR-30-1	VM 595	700 (371)	280
XSR-30-2	VM 595	750 (399)	240
XSR-31	VM 595	800 (427)	220
XSR-33	VM 615	700 (371)	280
XSR-35	VM 615	750 (399)	265
XSR-36	VM 617	700 (371)	250
XSR-37	VM 617	750 (399)	245

\*Break Through Tonage

TABLE 6. COMPOSITION OF ALLOYS EXTRUDED

<i>VM No.</i>	<i>En</i>	<i>Co</i>	<i>Al</i>	<i>Mg</i>	<i>Cu</i>	<i>Cr</i>
578	5.8	—	Bal	2.5	1.6	0.3
581	5.8	0.8	Bal	2.5	1.0	—
582	5.8	1.6	Bal	2.5	1.0	—
583	5.8	3.2	Bal	2.5	1.0	—
595	7.0	0.8	Bal	2.5	1.0	—
615	7.0	3.2	Bal	2.5	1.0	—
617	8.4	1.6	Bal	2.5	1.0	—

TABLE 7. SWAGING SCHEDULE FOR XSR 30-1

<i>Initial Size, in. (cm)</i>	<i>Final Size, in. (cm)</i>	<i>Reduction in Area %</i>
0.760 (1.930) <sup>1</sup>	0.720 (1.829)	10.2
0.720 (1.829) <sup>1</sup>	0.665 (1.689)	14.7
0.665 (1.689) <sup>1</sup>	0.590 (1.499)	21.3
0.590 (1.499) <sup>2</sup>	0.520 (1.321)	22.3
0.520 (1.321) <sup>2</sup>	0.466 (1.184)	19.7
0.466 (1.184) <sup>2</sup>	0.425 (1.080)	16.8
0.425 (1.080) <sup>2</sup>	0.390 (0.991)	15.8

<sup>1</sup>4 Jaw Swaging

<sup>2</sup>2 Jaw Swaging

## SECTION IV

### MATERIALS EVALUATION

#### ALUMINUM ALLOYS

Due to difficulty encountered in producing Al buttons, only one sample B-4 was treated by electron beam (EB) surface irradiation. This sample was given two EB passes to depths of 0.015 in. (0.038 cm) and 0.031 in. (0.079 cm). The results of this treatment look promising and a series of alloys will be evaluated in the next quarter. Figure 1 shows a representative weld and resulting microstructure.

Metallographic examination of all Al extrusions was completed in both the heat-treated and as-extruded conditions.

The heat treatment was 870°F (466°C) for 1 hr with a water quench plus 250°F (121°C) for 26 hr followed by an air cool which is the T-6 treatment for 7075 Al. This selection was made because of similarities between the XSR alloys and 7075 composition. The only difference is the XSR alloys have higher Zn contents and substitute Co for Cr. Since Co was not expected to contribute to precipitation hardening, the T-6 treatment was selected for initial studies. Representative microstructures appear in Figures 2 through 4.

In addition to the microstructural evaluation, hardness measurements were made after each step of the heat treatment. Results are tabulated in Table 8.

Microstructural evaluation and hardness testing were done on the three swaged samples, as shown in Figures 5 and 6 and Table 9, respectively. In all cases the hardness of swaged fully heat-treated samples displayed an improvement in hardness over as-extruded FHT samples. Additionally, the hardness values exceeded that of 7075-T6. Analysis of swaged microstructures showed a significant increase in grain size of the FHT structure when compared to FHT extruded material. Additional work by swaging probably breaks up a surface oxide which releases grain boundaries allowing grain growth. The surface oxide present was due to the handling of Al powder in air.

#### IRON ALLOYS

Considerable difficulty was encountered in heat treatment of Fe experimental alloys. Decarburization of EB welds did not allow for proper evaluation by microhardness, which is strongly dependent on carbon content. A vacuum furnace with a forced argon flow will be used to prevent decarburization of experimental alloys in further work.



Mag: 100X  
Etch: 20% Kellers

a.

FAM 89112



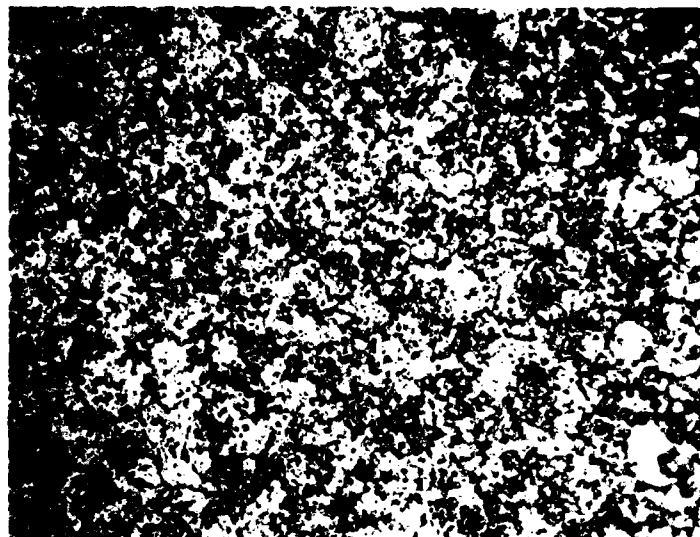
Mag: 400X  
Etch: 20% Kellers

b.

FAM 89113

FD 151988

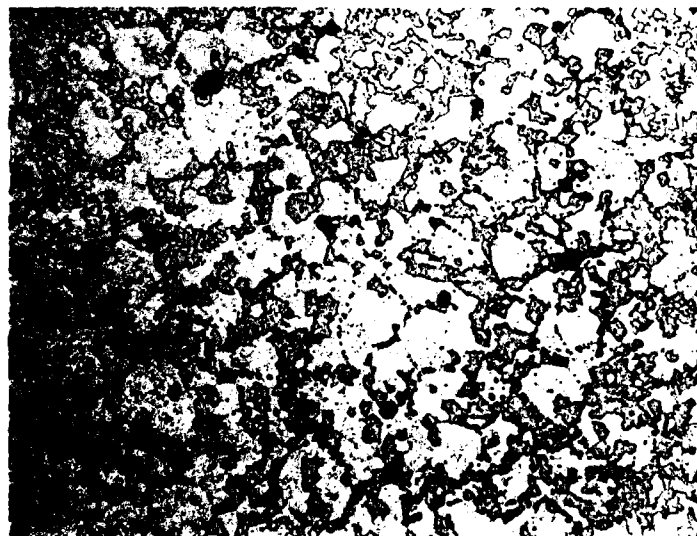
*Figure 1. Aluminum Button B-4 as EB Welded; Penetration 0.015 in. (0.038 cm)*



Mag: 400X  
Etch: Kellers

a.  
As-Extruded

FAM 89115



Mag: 400X  
Etch: Kellers

b.  
Heat-Treated

FAM 89114

FD 151989

*Figure 2. Microstructure of XSR 30-1*

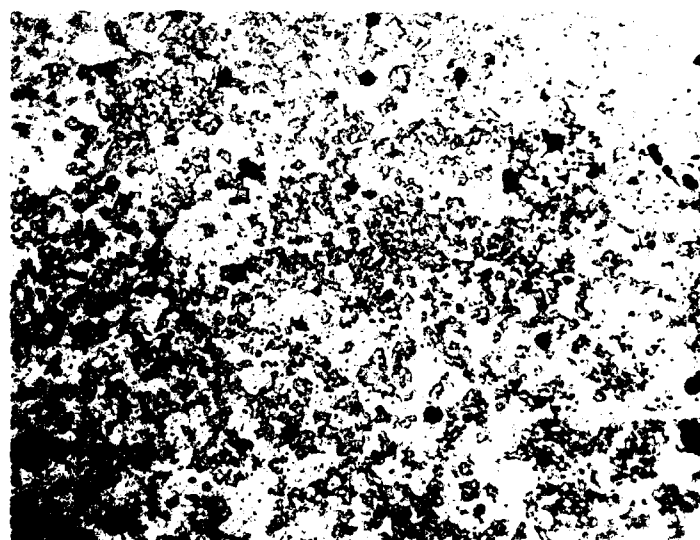




Mag: 400X  
Etch: Kellers

a.  
As-Extruded

FAM 89116



Mag: 400X  
Etch: Kellers

b.  
Heat Treated

FAM 89117

FD 151980

*Figure 3. Microstructure of XSR-33*



Mag: 100X  
Etch: Kellers

a.  
Longitudinal

FAM 89-18



Mag: 100X  
Etch: Keller

b.  
Cross Section

FAM 89-18

FT 100

*Figure 4. Microstructure of Conventionally Processed Al 7075-T6*

TABLE 8. HARDNESS OF ALUMINUM EXTRUSIONS, R<sub>H</sub>

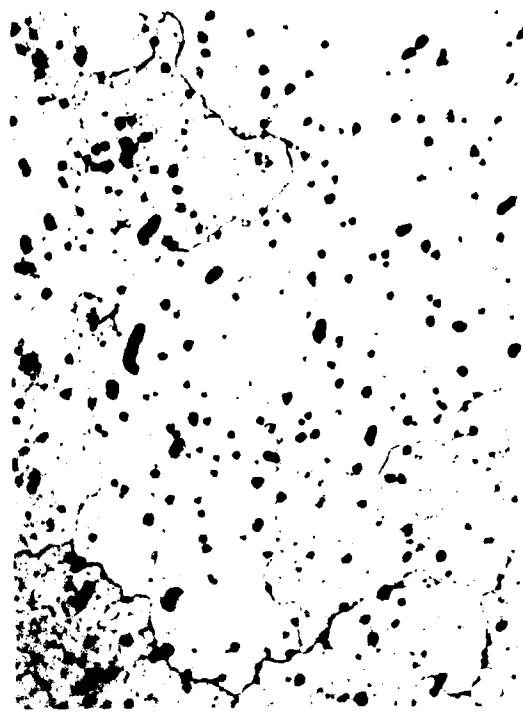
Sample No.	As Extruded	870°F(466°C)/1 hr/WQ +	
		870°F(466°C)/1 hr/WQ	250°F(121°C)/26 hr/AC*
XSR-19	21	61.8	49.3
XSR-20	26	64.0	48.7
XSR-23	47	73.5	83.5
XSR-25	46	73.7	82.8
XSR-27	53	73.8	88.5
XSR-30-1	50	68.0	85.8
XSR-30-2	52	72.8	82.8
XSR-31	43	67.7	83.0
XSR-33	54	77.3	91.5
XSR-35	51	72.0	81.8
XSR-36	53	69.8	86.3
XSR-37	54	74.8	86.3
7075	—	—	90.0

\*T6 Heat treatment



Mag: 400X a.  
Etch: Kellers As-Swaged

FAM 89123



Mag: 400X b.  
Etch: Kellers Heat Treated

FAM 89122



Mag: 100X c.  
Etch: Kellers Heat Treated

FAM 89120

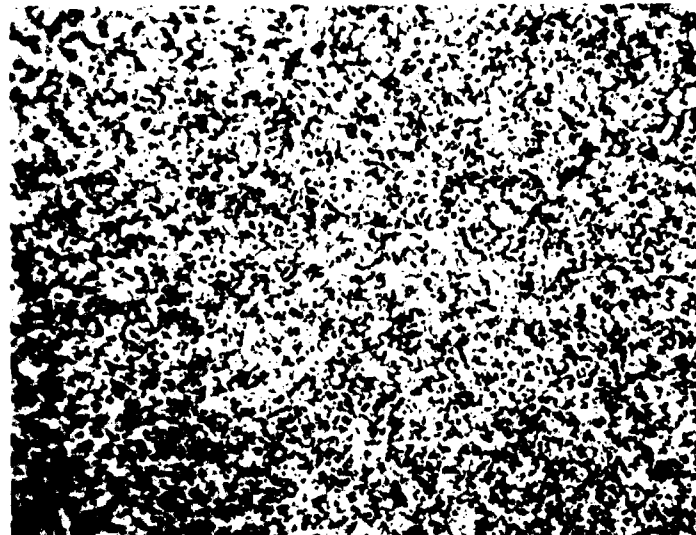


Mag: 100X d.  
Etch: Kellers Heat Treated

FAM 89121

FD 151992

*Figure 5. XSR 30-1 Extruded and Swaged*



Mag: 400X  
Etch: Kellers

a.  
As-Swaged

FAM 89124



Mag: 400X  
Etch: Kellers

b.  
Heat Treated

FAM 89125

FD 151983

*Figure 6. Microstructure of XSR-33 Extruded and Swaged*

TABLE 9. HARDNESS OF SWAGED  
ALUMINUM,  $R_H$

<i>Sample No.</i>	<i>As Swaged</i>	<i>FHT<sup>a</sup></i>
XSR-30-1	53.4	90.3
XSR-33	41.2	92.5
XSR-36	35.3	90.2

<sup>a</sup> FHT is 870°F(466°C)/1 hr/WQ + 250°F(121°C)/  
26 hr/AC

## **SECTION V**

### **ON-GOING STUDY**

Evaluation of the twelve Al extrusions will continue including mechanical testing. Hardness measurements and compressive yield testing will be done on the steel extrusions. Additional Al and Fe conversions will be made. There should also be several extrusions done at AFML of both Al and Fe compositions. Analysis of Al and Fe experimental samples produced during this report will continue with additional effort given to heat-treating the Fe samples. The new high-temperature furnace utilized in AGT 500,000 will be further evaluated for optimization of yield.

END

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